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**SPECTRAL STUDIES OF PROPELLANT COMBUSTION:  
EXPERIMENTAL DETAILS AND EMISSION RESULTS  
FOR M-30 PROPELLANT**

JOHN A. VANDERHOFF

DECEMBER 1988

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| <p>A windowed strand burner has been constructed and used to characterize the steady state burning of propellants at moderate pressure using uv - visible spectroscopic techniques. Emission spectroscopy has been performed on a triple base propellant (M-30) and three reactive transient combustion species (CN, NH and OH) have been profiled over a pressure range from 0.66 to 1.5 MPa. Reaction zone lengths on the order of 1 mm were determined from the CN and NH profiles. The strand burner is expected to be useful for future planned absorption and laser induced fluorescence studies on a variety of propellants.</p> |           |   |  |   |   |
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## I. INTRODUCTION

There is considerable uncertainty about the chemical and physical processes occurring in solid propellant combustion. Detailed probing of the gas phase region adjacent to the burning surface of the propellant is necessary for a more complete understanding of the combustion process. Spectroscopic experiments which identify and track the behavior of intermediate combustion products can provide information for developing reaction networks which assist in the characterization of the combustion chemistry.

Many articles have been written concerning the detailed chemical processes occurring in solid propellant combustion and two review articles by Fifer<sup>1</sup> and Lengelle, et al.,<sup>2</sup> well describe the important contributions. When considering only the spectral studies that employ visible and/or uv radiation the publication list is much smaller. Double base propellants have been studied with emission and absorption techniques.<sup>3,4</sup> In these studies blackbody temperature estimates, line emission signals from impurity metallic species and a  $C_2$  radical species were reported for the final flame zone (explosion zone). Ammonium perchlorate (AP) and nitramine propellants (HMX - cyclotetramethylenetetranitramine and RDX - cyclotrimethylenetrinitramine) have been studied more extensively in the recent past. Emission intensity profiles of several chemically active transient species have been obtained for AP and nitramine propellants.  $CN^{5-7}$  and  $OH^{6,7}$  profiles were measured for both types of propellants whereas an NH profile is published only for a nitramine propellant.<sup>6</sup> Equilibrium may not be achieved during combustion; thus, energetic species which emit visible/uv radiation are not necessarily equilibrated with respect to the partitioning of energy in their electronic, vibrational and rotational energy levels. Consequently emission intensity profiles are not always good indicators of actual species concentration. Nonetheless there is such a scarcity of actual data on transient species occurring in propellant combustion (absolute concentrations are virtually nonexistent) that the shortcomings of the emission technique are not yet a serious drawback. A number of techniques which optically probe the region of interest have a better potential for determining absolute concentrations. Laser induced fluorescence (LIF) measurements have been published which illustrate fluorescence intensity profiles of  $CN$  and  $OH^8$  in AP propellant combustion and of  $CN$ ,<sup>8,9</sup>  $NH$ ,<sup>9</sup>  $OH$ ,<sup>9</sup> and  $NO_2$ ,<sup>9</sup> in nitramine propellant combustion. A gas phase temperature profile has also been determined from the rotational structure of  $OH$ .<sup>9</sup> Coherent anti-Stokes Raman scattering (CARS)<sup>10</sup> has been applied to a nitramine propellant burning in room air. Several transient species were identified ( $HCN$  and  $NO$ ) and a two point profile with concentration estimates has been determined.

In this paper M-30, a common triple base propellant used for long range artillery and tank rounds, has been chosen for the initial studies. See Table 1 for the composition. Several reasons led to this choice of propellant. First, it has no apparent dark zone; thus the flame sits close to the propellant surface providing one local-hot region for spectral emission of intermediate species. Second, cylindrical sticks of this propellant were available. Spectroscopic studies have been carried out in a windowed strand burner. Spatially resolved uv-visible emission spectra for M-30 burning in a nitrogen environment have been obtained over a pressure range from 0.35 to 1.5 MPa. Species identification, spatial profiles of  $CN$ ,  $NH$  and  $OH$  and a

blackbody temperature estimate have been extracted from these emission spectra. Moreover, estimates of reaction zone lengths have been made from NH and CN emission intensity profiles. These results are then compared with other published reaction zone length values.

Table 1. Propellant Ingredients

| <u>Composition Wt. %</u> | <u>M-30</u> | <u>AP</u> | <u>API</u> | <u>HMX1</u> | <u>HMX2</u> |
|--------------------------|-------------|-----------|------------|-------------|-------------|
| Nitroquanidine           | 47.7        |           |            |             |             |
| Nitrocellulose           | 28.0        |           |            |             |             |
| Nitroglycerin            | 22.5        |           |            |             |             |
| Ethyl Centralite         | 1.5         |           |            |             |             |
| Sodium Aluminum Fluoride | 0.3         |           |            |             |             |
| Ammonium Perchlorate     |             | 78        | 87         |             |             |
| Polybutadiene Binder     |             | 22        | 13         |             |             |
| HMX                      |             |           |            | 73          | 80          |
| TMETN*                   |             |           |            | 17          |             |
| Polyester Binder         |             |           |            |             | 20          |

\*Trimethylolethanetrinitrate

## II. EXPERIMENTAL

An assembly drawing of the windowed strand burner constructed and used for the data reported here is shown on Figure 1. The goal of this design was to produce a compact vessel that would be versatile and time efficient. It is composed of four distinct parts: a base plate, main chamber, top piece and five window assemblies. All of these pieces screw rather than bolt together thus minimizing the time it takes to change propellant samples and windows. A video camera, used for burn rate measurements, eliminates the need for the time consuming operation of installing fuse wires in the propellant samples. This vessel was constructed from 304 stainless steel and has an overall height of 25 cm. It has an outside diameter of 15 cm and an inside diameter of 7.6 cm. The internal volume is approximately 0.9 liters. Five optical ports with a 2.5 cm clear aperture are available for use. Depending upon the experiment either normal incidence or Brewster's angle windows can be installed. The four ports which are opposed are currently used for experiments. The fifth port, located at the top, is for possible future use in radiation-augmented burning studies. To have the propellant burn in a cigarette fashion a flow of a nonreactive gas (nitrogen) around the propellant stick is required. The route of regulated nitrogen through the flow straighteners and out the exit orifice is illustrated on Figure 2. A 0.64 mm exit orifice was used. This provided a nitrogen flow rate of about four times the propellant gasification rate over the pressure range studied. Possible pressure transients which may occur during a propellant burn are minimized by the four liter surge tank plumbed to the strand burner. Ignition of the propellant, not shown, is accomplished by rapidly heating a small diameter nickel wire which has been placed on the propellant surface. The helium-neon (HeNe) laser is used for several purposes: optimizing the collection optics used for gathering the emission signals, measuring the transparency of the combustion gas and determining the position of the burning propellant surface.

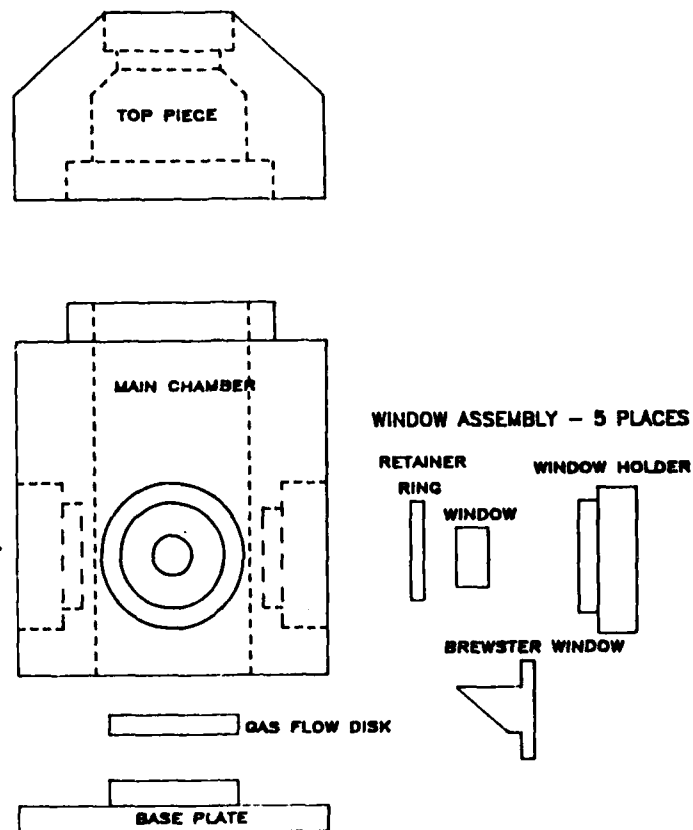


Figure 1. Strand Burner Assembly Drawing

A top view of the strand burner and associated optical systems are shown on Figure 3. Burn rates of the propellant samples are obtained with a video camera and macro lens. These video records also verify whether the propellant burns in a cigarette fashion with a horizontal, flat burning surface. Emission signals from the propellant sample are collected and focussed onto a 0.1 mm horizontally oriented entrance slit by two convex lenses with a magnification of two. The resulting spatial resolution is 0.1 mm full width at half maximum (FWHM). This resolution has been experimentally verified by recording the signal produced from various size illuminated slits as a function of position. A 0.25 m monochromator disperses the emission signal onto an intensified linear photodiode array and a computer is used for acquisition, manipulation and storage of the optical data. For the emission spectra taken here a 1180 grooves per mm grating has been used in the monochromator. This combination gives a spectral resolution of about 0.3 nm.

In a typical experiment the following sequence of events would occur. The propellant sample and ignition wire are installed in the strand burner. After closing the vessel it is then pressurized to the desired pressure and the video recording system turned on. Propellant burning is now initiated by

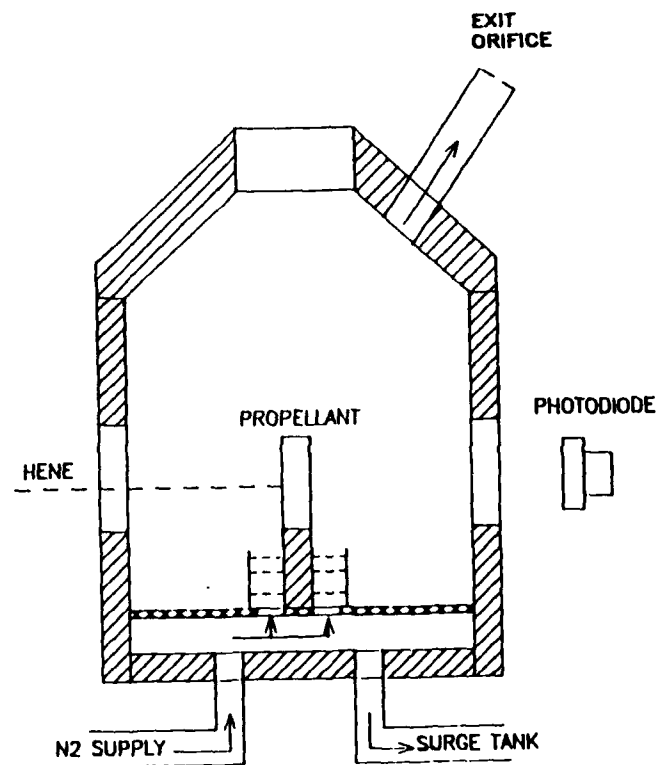


Figure 2. Cross Section of Strand Burner

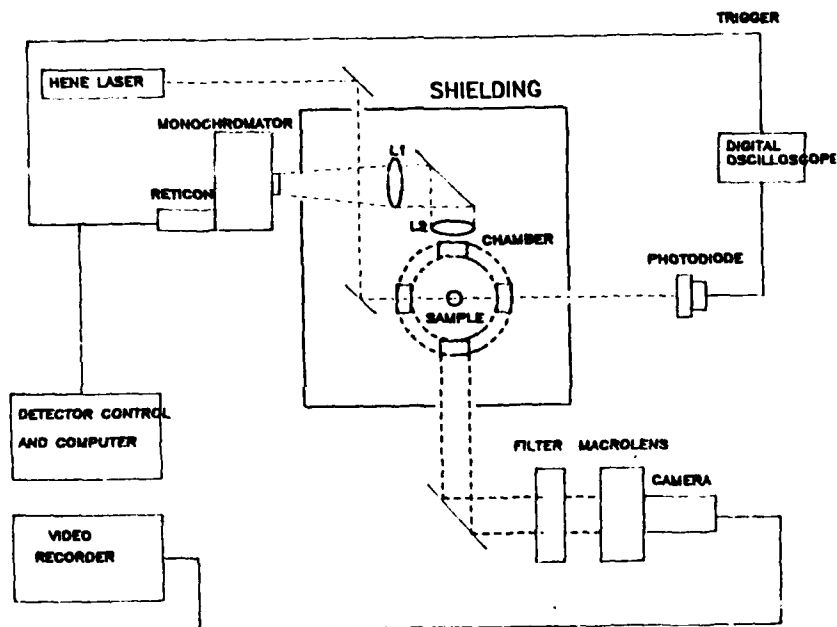


Figure 3. Top View of Strand Burner and Associated Optical Systems

switching on a power supply to heat the ignition wire. This switch also triggers a digital oscilloscope which records the HeNe photodiode output. Two methods have been used to initiate emission spectrum accumulation by the photodiode array. The simpler method is a manual start performed by the experimenter. The video camera real-time display provides the experimenter with a rough idea of when to start. Alternatively, the change in the HeNe photodiode output that occurs when the HeNe laser impinges on it has been used as an automatic start for emission spectra accumulation. Unfortunately this triggering method misses part of the data where the propellant surface is just entering the sampling volume but has not burned down to a position where there is no solid blocking the path of the HeNe. Once the photodiode array is triggered it repetitively scans and resets 120 times while storing each of these 120 scans into buffer memory. The total data accumulation time (typically 5 to 10 s) as well as the time for each scan can be varied over a wide range. An example of a portion of the multiple emission spectra obtained during a single propellant burn is shown on Figure 4. In this case the time used for each scan was 16 ms and the time between active scans was 48 ms; thus the emission was sampled every 64 ms. In the wavelength region shown on Figure 4 the time variation for CN (388 nm region or equivalently 3880 angstroms region) and K (405 nm) are represented for M-30 burning in 0.66 MPa (6.6 atm) of nitrogen. This temporal variation of the emission spectra can be converted to a distance from the propellant surface by using the burn rate of the propellant as determined from the video record. Emission intensities for individual species, calculated by integration of their spectral features, can be determined as a function of distance from the propellant surface. It should be noted that the CN and NH (336 nm) species are relatively short lived; that is, they appear close to the propellant surface and typically are consumed at distances less than 2 mm from the burning surface. Considering this small extent, a shadowing effect occurs which can alter the emission intensity profile; see Figure 5. Previous investigators have discussed this effect<sup>5,8,11</sup> and in one case a trial and error method<sup>5</sup> was used to extract a "corrected" profile. Since future experiments to measure the transient species involve a technique (optical absorption) that is not influenced by shadowing, no significant attempt has been made to account for shadowing. A final comment about this effect is that it goes in a direction that makes the intensity profiles appear narrower than the actual case. This observation, together with a comparison of the present data for NH and CN with the published data of Branch, et al.,<sup>12</sup> on a low pressure flame suggests one possible correction. The low pressure flame data look much more symmetric than the present data which have a rather abrupt rise. If this abrupt rise were altered to be symmetric with the fall off, the FWHMs reported here for CN and NH would be increased by about 25%. Non-horizontal burning of the propellant surface will also compromise the spatial resolution of the emission intensity profiles. The video record has a resolution of about 0.15 mm for the detection of this effect and provides a quality control on acceptable propellant strand burns. Lastly, the propellant surface moves during the data accumulation and readoff of the photodiode array. For these experiments the scan time was 16 ms and the burning rate varied from about 1 to 3 mm/s depending on the pressure. Thus the burning surface moves at most 0.05 mm during an emission scan.

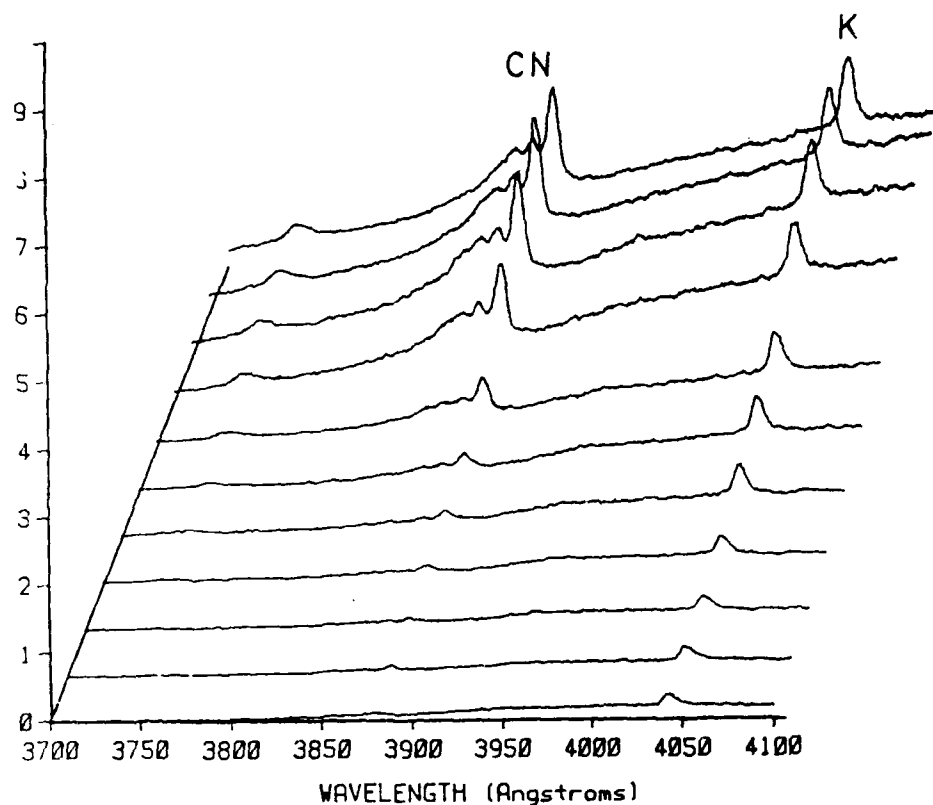


Figure 4. Multiple Emission Spectra Versus Wavelength.  
Here only a portion of the 120 emission spectra taken for each  
propellant burn is shown. These spectra are 64 ms apart in time.

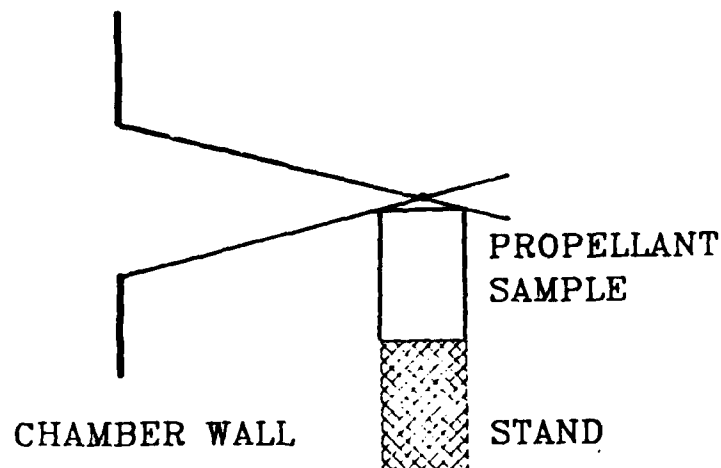


Figure 5. Detail of the Optical Collection System Showing the Geometry  
that can Cause a Shadowing Effect. The angle of collection has been  
exaggerated for clarity. At positions where the propellant surface is  
closer to the optical focus point of the collection lens part of the  
light emission will be blocked from view by the propellant.

### III. RESULTS

One of the more important measurements for characterizing a propellant is the burn rate as a function of pressure. Moreover, this parameter is required in the present context to be able to plot emission intensity as a function of distance. The propellant samples for study are small solid cylinders 6 mm in diameter and 20 mm long. To minimize ignition effects and different temperature gradients close to the propellant strand stand, only the middle 10 mm was observed for burn rate measurements. A macro lens magnified the propellant strand nine times and the video record of the burn enabled the burn rate to be determined with an estimated absolute accuracy of  $\pm 8\%$ . A comparison of the present results with unpublished data of Miller<sup>13</sup> is shown on Figure 6 and the agreement is excellent.

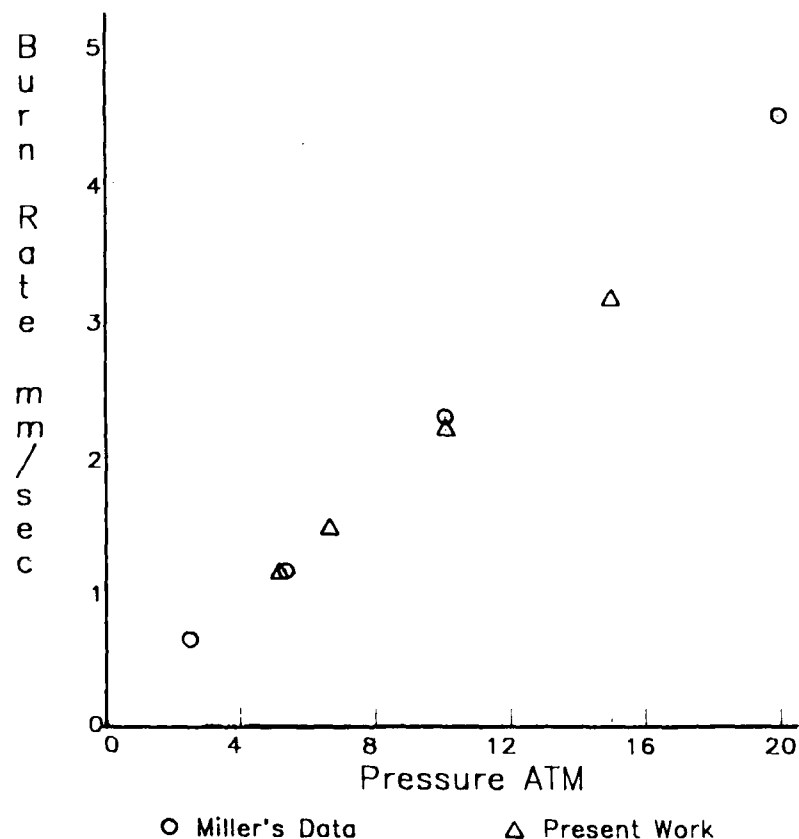


Figure 6. A Comparison of Burn Rate Data for M-30 Propellant at Moderate Pressures

A coarse emission spectrum for M-30 burning at 0.6 MPa (6.6 atm) is shown on Figure 7. The wavelength range extends from 300 to 680 nm. The emission detection system could only capture approximately 50 nm of spectra at a time so Figure 7 is a result of piecing together eight individual spectra. A standards lamp was employed to calibrate the detection system for wavelength sensitivity and geometric effects; hence the emission intensity is an accurate relative intensity profile with small line emissions omitted. Enormous sodium D lines emissions are observed which dominate the detection system for more than a 50 nm range. The small amount of sodium aluminum fluoride used as a muzzle flash suppressant has produced the overwhelming majority of visible

emission. A portion of the spectrum removed from the sodium line emission is shown in greater detail on Figure 8. Assuming the emission to be dominated by continuum blackbody radiation and the emissivity to be wavelength independent over this range a temperature characteristic of the combustion gas can be calculated from Planck's radiation equation. A least squares fit to the data results in a temperature of 2587 K with a standard deviation of 189 K. This temperature is less than the calculated adiabatic value of 3040 K; this is not too surprising since the measurements were made at 0.66 MPa (6.6 atm) where it is probable that the chemistry has not reached completion. What is assumed in the above analysis is that continuum chemiluminescence is negligible. This means concentrations of common combustion species such as  $\text{NO}_2$  and combustion reactions like  $\text{CO} + \text{O}$  that exhibit broad band chemiluminescence over this wavelength region are neglected. In some cases this is certainly not true; i.e., close to the propellant surface where these species can exist. On the positive side the data of Figure 8 are evenly distributed about the least squares fit which means that the emission data have a wavelength dependence characteristic of blackbody emission. A serious attempt to obtain gas phase temperature profiles using this technique should employ a much coarser grating so that at any given time a larger portion of the wavelength spectrum would be sampled.

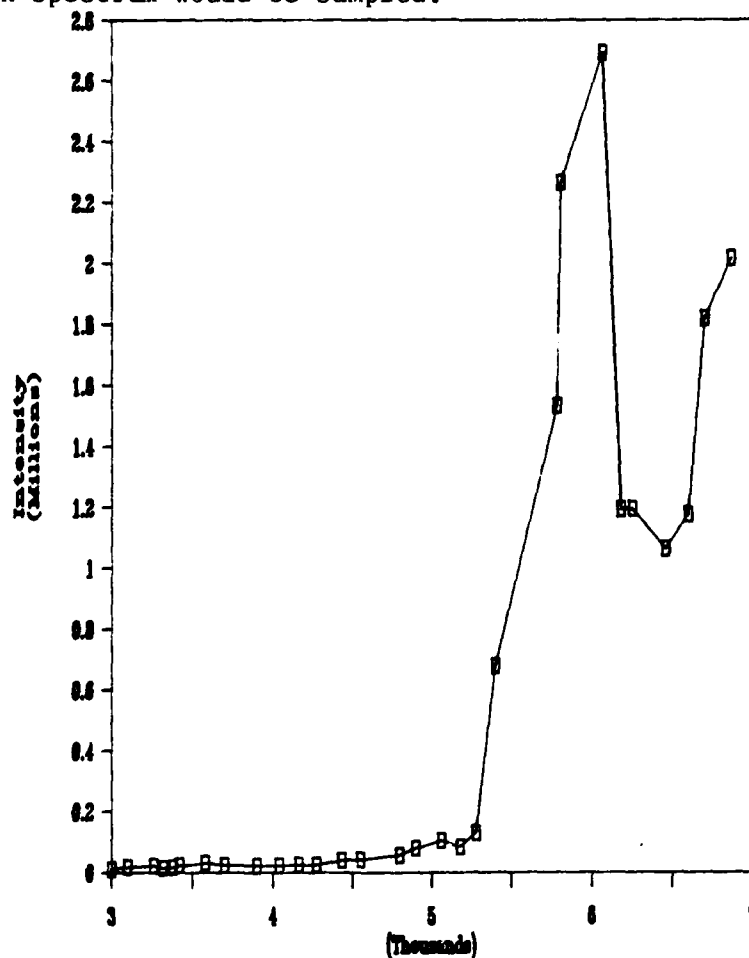


Figure 7. Calibrated Emission Intensity Versus Wavelength from 300 to 680 nm for M-30 Burning at 0.66 MPa. Only the overwhelming line emission from the sodium D lines have been included in this coarse emission spectrum.

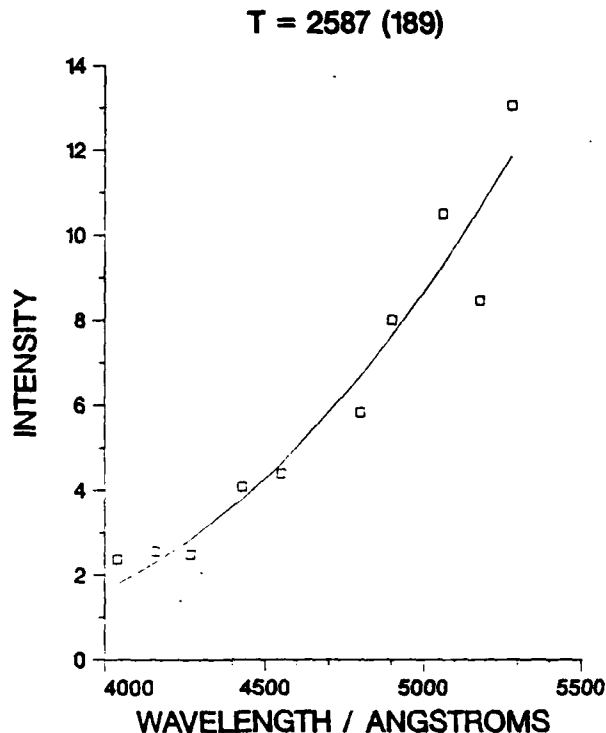


Figure 8. Calibrated Emission Intensity Versus Wavelength Over a Much Reduced Range, 390 to 530 nm. This profile is a subset of the profile of Figure 7. The solid line is a least squares fit to the data to extract a blackbody temperature.

Spectroscopic observation of the 388, 336, and 307 nm wavelength regions provided the necessary emission data to allow spatial profiling of CN, NH, and OH over a pressure range from .35 to 1.5 MPa. Representative emission spectra from the region which includes CN, NH, and OH emission are shown on Figures 9a, b, c, respectively. Multiple emission spectra are used to obtain the CN, NH and OH emission intensity versus distance from the propellant surface. These profiles are shown on Figures 10, 11, and 12, respectively. The negative distances indicate that the propellant has not burned past the detection volume. Reaction zone lengths have been determined from the FWHMs of these types of profiles and the results together with other published values<sup>5,6,8,9</sup> for other propellants are listed in Table 2. The propellant ingredients are listed in Table 1. There is such a scarcity of reaction zone length data for solid propellant combustion that the data in Table 2 include two formulations of ammonium perchlorate (AP and AP1) which are composite propellants, three kinds of nitramine propellants (HMX - neat; HMX1 and HMX2 - composite) and a triple base propellant (M-30). HMX and HMX2 are reported to have dark zones while HMX1, AP, and M-30 do not. These and other differences in the various propellants studied make quantitative comparisons of dubious value and point out a need for experimental efforts to focus on a few standard propellants. Qualitatively, the values of Table 2 generally agree to well within a factor of three and the reaction zone lengths are mostly larger than that of a simple atmospheric pressure flame (0.5 mm). In the present study it

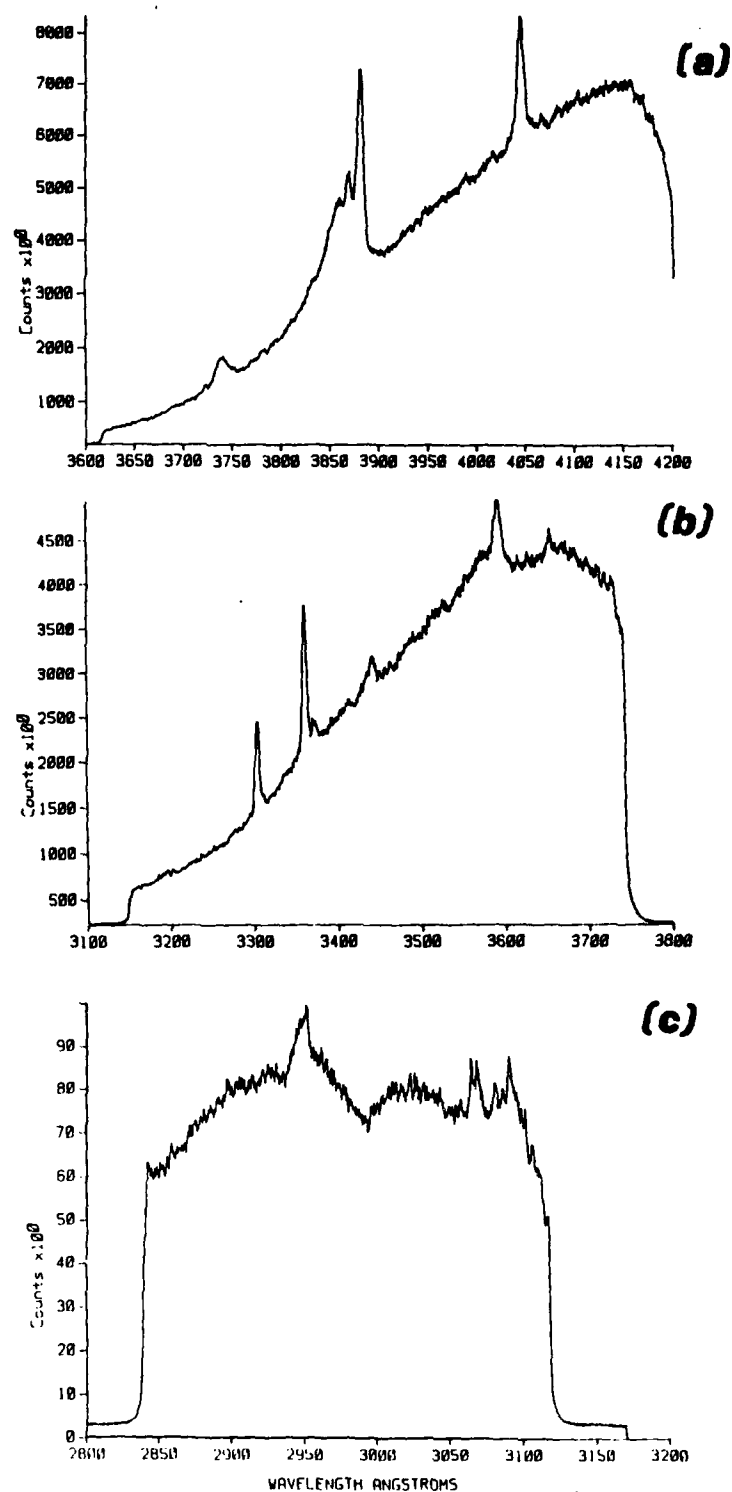


Figure 9. Emission Intensity Versus Wavelength for M-30 Burning at 0.66 MPa. These spectra have not been corrected for either wavelength sensitivity or geometric effects. (a) CN emission is readily observed over the region from 385 to 390 nm. Potassium emission is detected at 405 nm. (b) NH emission is observed at 336 nm, CN at 359 nm, and Na at 330 nm. (c) OH emission is observed in the 305 to 310 nm region. A prominent peak at about 295 nm has not been identified.

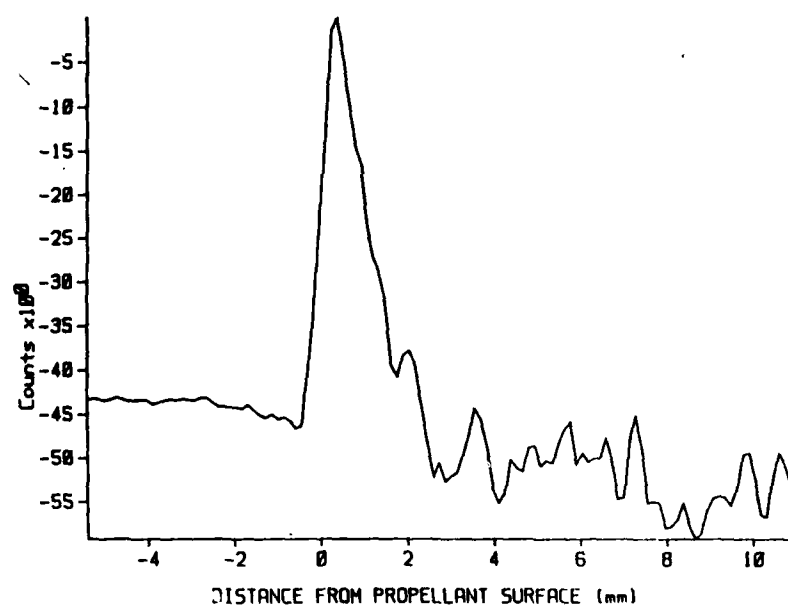


Figure 10. Emission Intensity for CN Versus Distance from the Propellant Surface Using M-30 Propellant Burning at 1.0 MPa

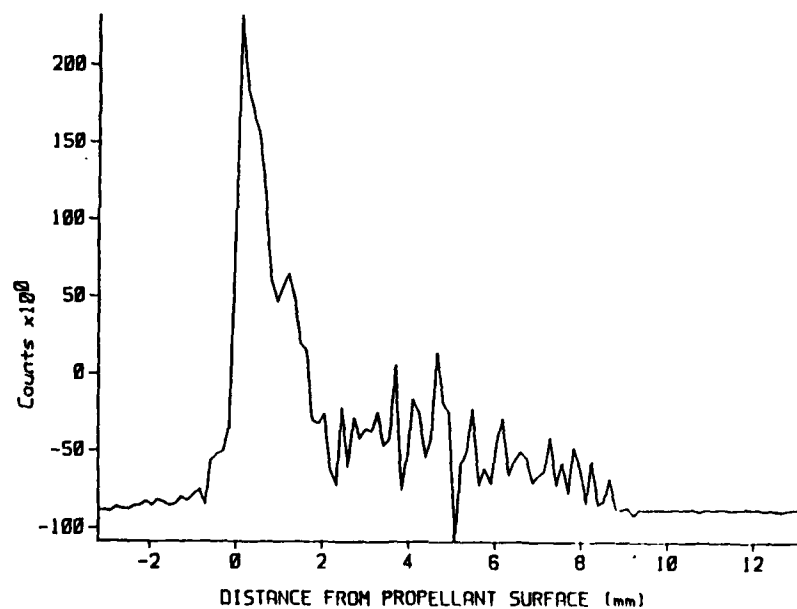


Figure 11. Emission Intensity for NH Versus Distance from the Propellant Surface Using M-30 Propellant Burning at 1.0 MPa

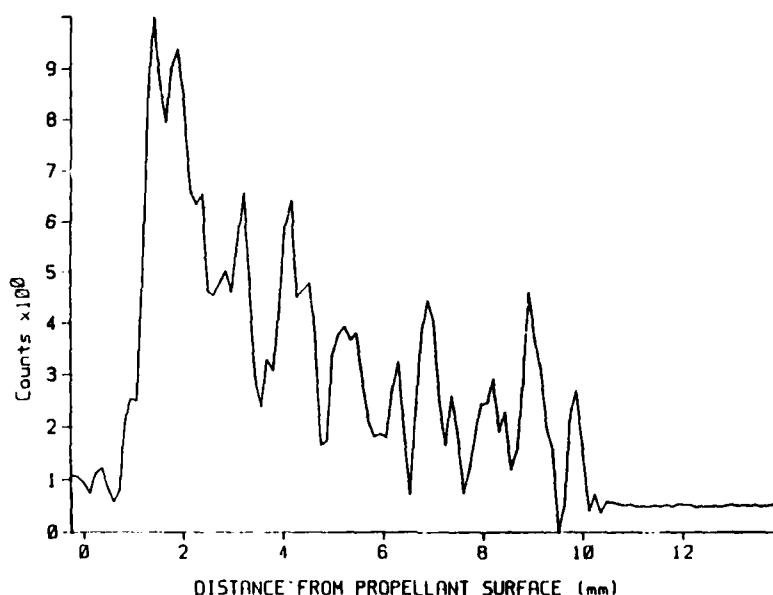


Figure 12. Emission Intensity for OH Versus Distance from the Propellant Surface Using M-30 Propellant Burning at 1.0 MPa

Table 2. Summary of Reaction Zone Lengths for Various Propellants

| <u>Species</u> | <u>Reaction Zone Length</u> | <u>Pressure</u> | <u>Propellant</u> | <u>Investigator &amp; Technique</u> |
|----------------|-----------------------------|-----------------|-------------------|-------------------------------------|
| CN             | 2.0 mm                      | 1 atm           | AP                | Povinelli - Emiss                   |
| CN             | 2.5 mm                      | 1 atm           | AP1               | Edwards - Emiss                     |
| CN             | 0.7 mm                      | 1 atm           | HMX               | Parr - PLIF                         |
| CN             | 0.6-0.4 mm                  | 1-8 atm         | AP1               | Edwards - LIF                       |
| CN             | 0.5 mm                      | 7-35 atm        | HMX1              | Edwards - LIF                       |
| CN             | 1.8-0.5 mm                  | 18-35 atm       | HMX2              | Edwards - LIF                       |
| CN             | 1.5-1.2 mm                  | 6-15 atm        | M-30              | Present work - Emiss                |
| NH             | 0.5 mm                      | 1 atm           | HMX               | Parr - PLIF                         |
| NH             | 1.2 mm                      | 35 atm          | HMX1              | Edwards - Emiss                     |
| NH             | 1.0-0.7 mm                  | 6-15 atm        | M-30              | Present work - Emiss                |

has also been observed that to about 0.1 mm both NH and CN appear at the same place but CN extends further out in the propellant combustion gas. The data that supports this conclusion comes from the larger reaction zone lengths observed for CN and from the data of Figure 13. In order to produce the data in Figure 13 the spectrometer was set analogous to that for recording the emission spectrum shown on Figure 9b. Here NH appears at 336 nm but now a vibrationally excited band of CN can be observed at 359 nm. This excited band of CN is much weaker than the 388 nm band but its emission intensity profile indicates that CN appears about 0.1 mm closer to the propellant surface than NH.

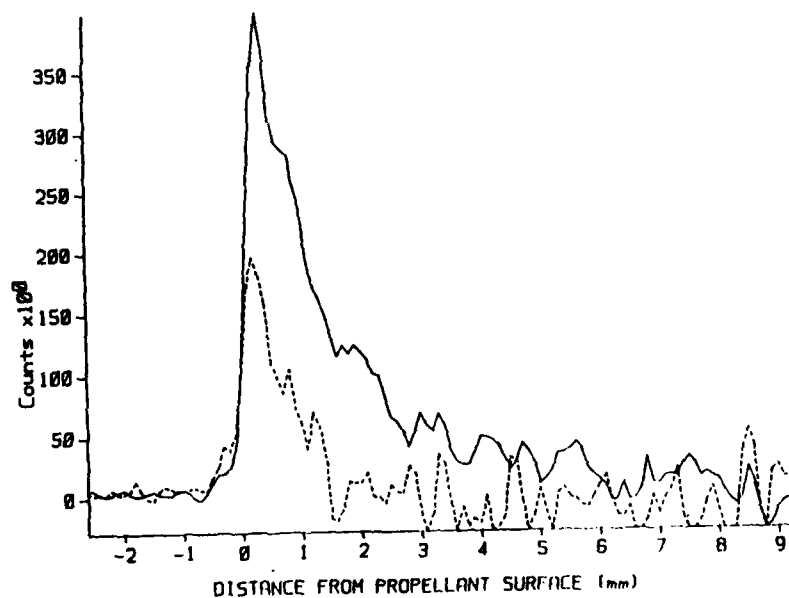


Figure 13. Emission Intensity for NH (Solid Line) and CN (Dotted Line) Versus Distance from the Propellant Surface. The emission intensities were recorded simultaneously for M-30 burning at 0.66 MPa.

Better signal-to-noise characteristics are observed for CN and NH emission profiles relative to the OH emission profile shown on Figure 12. Nonetheless, the OH emission intensity rises sharply and extends further out into the flame zone than does either CN or NH. This behavior is found in most flame systems.

#### IV. FUTURE WORK

Future studies include determining the applicability of employing broadband uv-visible absorption as a combustion diagnostic for these strand burner studies. Should absorption measurements prove feasible a variety of concentration profiles for intermediate combustion species could be obtained. With sufficient spectral resolution temperature profiles may also be extracted.

LIF is also planned to be used to profile intermediate combustion species such as NCO.

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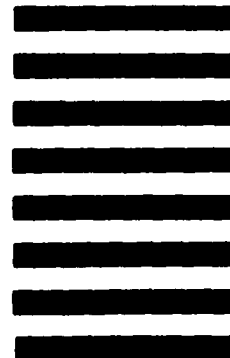
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